FSTC-HT-23-681-71

ARMY MATERIEL COMMAND

U.S. ARMY FOREIGN SCIENCE AND TECHNOLOGY CENTER

INVESTIGATION TECHNIQUE OF MICROHARDNESS OF HIGH MELTING POINT ALLOYS IN A WIDE TEMPERATURE RANGE

by

V. N. Skuratovskii
Iu. G. Tkachenko
V. A. Borisenko

USSR

This document is a rendition of the original foreign text without any analytical or editorial comment.

Approved for public release; distribution unlimited.

12

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Foreign Science and Technology Center US Army Materiel Command Department of the Army	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

INVESTIGATION TECHNIQUE OF MICROHARDNESS OF HIGH MELTING POINT ALLOYS IN A WIDE TEMPERATURE RANGE

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Translation

5. AUTHOR(S) (First name, middle initial, last name)

V. N. Skuratovskii, Iu. G. Tkachenko, V. A. Borisenko

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
27 JAN 72	12	N/A

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO.	FSTC-HT-23-681-71
c. T702301 2301	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d. Requester Avn Sys Command	OACSI No. J-9844

10. DISTRIBUTION STATEMENT

Approved for public release; distribution unlimited.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	US Army Foreign Science and Technology Center

13. ABSTRACT

The study of high-melting compounds, such as carbides, borides and nitrides, by the microhardness method is still an acceptable means of observing their static hardness over a wide range.

The purpose here is to consider certain methodological problems in the study of microhardness in these substances over a wide temperature range--the preparation of test samples, and the selection of load and of the time-parameters of loading.

DD FORM 1 NOV 68 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Microhardness						
Hardness						
Titanium carbide						
Boron carbide						
Metal compound						
Transition metal						
Solid mechanical property						
Material test						
Temperature Dependence						
Temperature						

TECHNICAL TRANSLATION

FSTC-HT-23-681-71

ENGLISH TITLE: INVESTIGATION TECHNIQUE OF MICROHARDNESS OF HIGH MELTING POINT ALLOYS IN A WIDE TEMPERATURE RANGE

FOREIGN TITLE: METODIKA ISSLEDOVANIYA MIKROTVERDOSTI TUGOPLAVKIKH SOYEKINENIY V SHIROKOM INTERVALE TEMPERATURE

AUTHOR: V. N. Skuratovskii, Iu. G. Tkachenko, V. A. Borisenko

SOURCE: Problemy prochnosti (Hardness Problems), No. 4, October 1969, Pages 74-78

Translated for FSTC by ACSI

NOTICE

UDC 620.178.5

INVESTIGATION TECHNIQUE OF MICROHARDNESS OF HIGH MELTING POINT ALLOYS IN A WIDE TEMPERATURE RANGE

Problemy prochnosti
(Hardness Problems),
No. 4, October 1969,
Pages 74-78

V. V. Skuratovskiy,
Yu. G. Tkachenko,
V. A. Borisenko

The study of high-melting compounds, such as the carbides, borides and nitrides, by the microhardness method is still an acceptable means of observing their static hardness over a wide temperature range. The method, moreover, offers a reliable index of the level of hardness of these compounds.

But tests of this sort entail considerable methodological difficulty, arising not only from the very high level of hardness present, but also from the brittleness of the substances. It is this factor, evidently, which has caused the relative lack of published data on static hardness in the presence of high or very high temperatures.

In the present article our purpose is to consider certain methodological problems in the study of microhardness in these substances over a wide temperature range — more precisely, the preparation of test samples, and the selection of load and of the time-parameters of loading.

Our tests were run with a unit designed by the Institute of Hardness Problems, Ukrainian Academy of Sciences [1]. The unit was intended precisely for testing microhardness over a wide temperature range. Our sample, measuring 8 mm in diameter and 4-5 mm in height, was placed on a stand within the hermetically sealed unit, and subjected to pressures of $1 \cdot 10^{-5} - 8 \cdot 10^{-5}$ mm Hg during the course of the experiment. By applying thermal radiation from a tungsten heater, both sample and indenter were heated to the same temperature. The temperature in the unit was controlled by a tungsten-rhenium thermocouple, which led through a cavity in the stand to the base

of the sample, where, for reliability of thermal contact, its soldered joint was pressed to the body of the sample by a pin. During the course of the test, temperature oscillation in no cases exceeded ± 2-3°C, and deviation from the norm was never more than 2%. The indenter was of the four-sided type, with apex angle of $136^\circ \pm 20'$. At room and very high temperatures (up to 1,000°C), diamond was the material used to prepare the indenter; at high temperatures (800-900°C and higher), boron carbide (B_4C). The diameters of the impressions made were measured with the MIM-8M metallographic microscope and an MOV-1-15 screw-type ocular micrometer; the magnification was 900.

The test samples were obtained by hot forging in graphite molds. In composition, the samples were almost stoichiometric. Their surfaces were first polished with ASO-16-B1-50 diamond wheels on a surface-grinding machine, then polished in turn on a 100.40-micron-and a 7-micron-grain elastic diamond disk. Electrolytic polishing was used to eliminate the formation of a cold-hardened zone during mechanical processing of the sample. In the case of carbides of the transition metals, the following composition of the electrolyte was used: 3 parts hydrofluoric acid (HF), 5 parts nitric acid (HNO_3), 3 parts acetic acid (CH_3COOH), and traces (a few drops) of bromine. Tantalum wire was used as a cathode. Voltage and current strength were 4.5 V and 1 A.

In testing for microhardness over a wide temperature interval an important factor is the choice of load to be placed upon the indenter. There are three basic conditions which must be observed in this connection. First, it is well known that following achievement of a certain imprint which is critical for the material being worked on, the observed microhardness begins to vary as the load is decreased. This phenomenon (in the authors' opinion) may be associated, on the one hand, with deviation, during the tests, from the law of mechanical similarity; and, on the other, with growth in the number of instrumental errors in the region of small loads.

In Table 1 are shown the results of measurements of the microhardness of titanium carbide, for various loads and temperatures: these results make it clear that with decrease in load there appears a tendency toward increase in microhardness, while at high temperatures this increase proceeds less intensively.

This leads to a definite conclusion: in the low temperature range the effect of factors resulting from instrumental errors and deviation from the law of mechanical similarity appears more prominantly, and as a result the character of the temperature relationship may be significantly distorted in cases where the load is chosen in the region of intensive variation in microhardness.

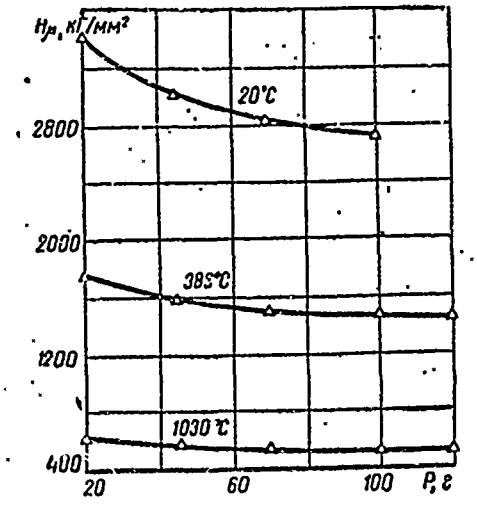

Figure 1. Variation in microhardness as affected by load; for TiC at various temperatures.

On the basis of this first condition, it is clear that the magnitude of the load should be placed higher in the case of temperatures lower than those used in our tests. But it cannot be shifted indefinitely — first, on account of requirements defining the ratio of the size of the impression to the micro-object under study; and, second, on account of the formation of cracks around the impression. As is well known, a correct determination of monocrystalline hardness requires that the volume of material being deformed under the indenter, if it is to avoid the effect of neighboring grains, boundaries and pores, must not go beyond certain limits, depending upon the dimensions of the structural component.

Measurement of the deformed zone shows that those dimensions exceed the diameter of the impression by a factor of approximately 2.6 [2]. On the surface of the sample this deformed zone is rather larger, thanks to the effect of the "weakened layer" [3]; for this reason we can assume that with variation in microhardness, the distance from the boundaries of the micro-object under study to the impression should be not less than 3d.

In the case the carbides of the transition metals (for the temperature range of our studies), the dimensions of the impressions are commensurate with those of the structural components only for high temperatures. Based on this, we should correct the magnitude of load for the maximal temptures of the range studied, in accordance with the foregoing.

As our tests show, carbides and borides maintain their high brittleness even at fairly low temperatures. In turn, this brings on the formation of cracks around impressions, and with any increase in load the process of crack-formation intensifies and begins to make itself felt on the magnitude of microhardness.

A quantitative evaluation of brittleness in materials, based on the degree of development of cracks close to impressions, has already been made [4, 5], using a "brittleness scale". Following this scale, and taking the character of the impression into account [5], the present writers believe that the optimal load is the one for which the character of the impressions produced does not transcend the bounds established in "brittleness Number 2". On this basis, at least, the effect of cracks on the magnitude of microhardness is quite absent: the impression maintains distinct outlines, and it can therefore be measured. The studies referred to were made with 900-power magnification.

TABLE 1

High-melting compound:	TiC	ZrC	HfC	NbC	TaC	WC	Mo_2C
Load, P_{max}, in g:	100	100	70	70	150	150	90

In Table 1 above are given the optimal loads for certain carbides of the transition metals which satisfy the condition referred to at room temperature.

On the basis of the foregoing, the magnitude of the load must be adjusted to suit lower temperatures within the range being studied.

In view of the limitations imposed by the conditions, a load of 60-80 g can be adopted for the study of titanium carbide over a wide range of temperatures.

In testing materials which closely resemble one another in mechanical properties (for example, the carbides of the Groups IV-VI of the transition metals), it is advantageous to strive for unification of the magnitude of load. It has been found experimentally that the load range of 60-80 g, established for titanium carbide, can also be used in testing the other carbides of those groups, provided the above-mentioned conditions be observed.

At the start of the loading process, the indenter is shifted up to contact with the sample, after which it penetrates the tested material, as the load increases. It is upon the magnitude of the time parameters which characterize this stage of the tests, that the results of the experiment depend. Assuming that the magnitude of microhardness must be such as to deflect the resistance of the material to plastic deformation, then a representative of this particular stage should be sought in a parameter which will directly define the process of indentation.

For this reason, the most characteristic parameter is growth rate of the load, which indeed characterizes the process of indentation, and remains constant over a wide range of temperatures. Observation of no more than one growth rate of load on various sets of apparatus assures identical conditions for the conduct of the tests, and in addition offers the possibility of comparing different sets of experimental findings. The means of securing a given growth rate naturally depends upon the loading apparatus in use in a given case -- it is a "local" thing. Still, there are certain physical factors which are the inevitable concern of every shop applying the "microhardness method": the forces of inertia within moving masses, which must somehow be excluded; and the fact that increase in the load growth rate intensifies the phenomenon of crack-formation around the impression, thus disrupting the process of plastic flow under the indenter -- just to mention two.

As has been shown by tests run on the most brittle materials derived from the group of carbides under consideration, NbC and HfC samples subjected to load growth rates in the interval 10-20 g/sec have exhibited no disruption of the process of plastic flow under the indenter. It follows that, to study the carbides of the transition metals to learn their behavior in the 20-1,800°C range, we may reasonably accept a loading rate of 15 g/sec.

Following application of full load, the indenter continues to penetrate the material the resisting force of the latter equalizes the applied load. Then the stabilized process of penetration, which is characterized by a constant rate of plastic flow in the material -- in other words, the variation rate of the diagonals, and, correspondingly, the rate of variation in microhardness, are stabilized.

As Kuznetsov [6] has pointed out, during the period of unstabilized penetration, the processes taking place under the indenter almost defy any explanation. It is obvious that the magnitude of microhardness, being one of the mechanical characteristics of the properties of materials, ought to be determined at the stage of stabilized penetration. In Table 2 below are given the results of measurement of the microhardness of NbC for various temperatures and periods under a load.

TABLE 2

Time under load, t, sec:	Microhardness at two temperatures:	
	20°C	1,690°C
5	1,975	—
15	1,900	—
20	1,900	137
30	1,900	132
40	—	130
50	—	130
60	—	129
90	—	129
100	—	128

At room temperature, the magnitude of microhardness becomes constant after the material has remained under the indenter for 5-10 sec. At high temperatures, the same thing happens after 20-30 sec. Similar results were obtained for TiC, TaC and WC. It follows from this that with the use of carbides at temperatures from 20 to 1,800°C, an underload time of 30 sec might be expected.

Load-withdrawal time, it is true, has less effect on the results of the experiment than do the other time factors. It should be noted, however, that with rapid rise of the indenter (in some machines this is accomplished with a magnet) there sometimes occurs a tearing away of the material, owing to the action of cohesive forces arising in the contact zone; this result is especially common in the case of high temperatures, with which a boron carbide identer is used. Unloading, then, should be carried out without sharp separations, provided, of course, this does not violate the avowed purposes of the research.

In our experiments, the time before the load was released, following automatic cutoff of the electric drive, was taken to be equal to the

time of loading. On the basis of the method described above, some research was undertaken on the temperature relationships of the microhardness of carbides, the individual results are shown below in Figure 2:

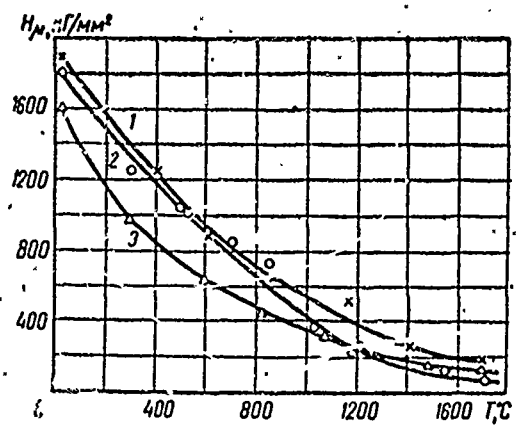

Figure 2. Temperature dependence of the microhardness of several carbides: 1 - NbC (threshold 7%); 2 - WC (thr. 2%); 3 - TaC (thr. 8%).

The stated parameters of tests for microhardness can be recommended for use in further research in high-melting compounds such as carbides and borides, tested over a wide range of temperatures.

BIBLIOGRAPHY

1. Pisarenko, G. S., Skuratovskiy, V. N. and Borisenko, V. A. "A Machine for the Study of Microhardness of Materials at High Temperatures"; Termoprochnost materialov i konstruktivnykh elementov (Thermal Strength of Materials and Design Elements), No. IV, Kiev, "Naukova Dumka", 1967.

2. Vatman, F. F. and Zlatin, N. A. Zavodskaya Laboratoriya (The Plant Laboratory), 1969, No. 4.

3. Shaposhnikov, N. A. Mekhanicheskiye ispytaniya metallov (Mechanical Tests of Metals), Moscow, Mashgiz, 1954.

4. Metody ispytaniya na mikrotverdost' (Methods of Testing by Microhardness), Moscow, "Nauka", 1965.

5. Glazov, V. M. and Vigdarovich, V. N. Mikrotverdost' Metallov (Microhardness in Metals), Moscow, Metallurgizdat, 1962.

6. Kuznetsov, V. D. Issledovaniye po zharoprochnym splavam (Research on Heat-Resistant Alloys), Vol. 5, Moscow, Publ. House of the USSR Academy of Sciences, 1959.

Institute of Strength Problems
Ukrainian Academy of Sciences

Received by Editor 21 Jan. 1969